

Lipophilic carbon quantum dots enable fluorescence-traceable lubricant release and tribofilm formation in self-lubricating fabric composites

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Abstract: Lubricant delivery and transfer-film formation govern friction and wear in enclosed sliding contacts, yet these buried interfacial processes are difficult to monitor. Oil-loaded microcapsules can supply lubricants to self-lubricating fabric composite liners, but their release behavior is usually optically silent. Here, we report an optically self-reporting microcapsule strategy for AF/PTFE fabric composites, in which dodecyl-chain-modified carbon quantum dots (CQDs-C₁₂) were dispersed in PAO4 and encapsulated in SiO₂ microcapsules as fluorescent lubricant reservoirs. During ball-on-disk sliding, local contact stresses triggered the release of CQDs-C₁₂/PAO4 into the wear track. The normalized integrated fluorescence area provided a semi-quantitative optical indication of the cumulative release and accumulation of the fluorescent lubricant phase. The optimized CD-MCs /Fabric achieved a friction coefficient of 0.085 and a wear volume of $1.03 \times 10^{-10} \text{ m}^3$, which were 61.7% and 44.5% lower than those of Pure Fabric, respectively. SEM/EDS, Raman, XPS and cross-sectional FIB-TEM analyses confirmed the formation of a continuous multi-component tribofilm on the steel counterpart, which reduced direct fabric/steel contact and interfacial shear. This work couples optical release reporting with tribofilm-mediated lubrication, providing a route toward observable self-lubricating fabric composites for enclosed sliding contacts.

KEYWORDS: Self-lubricating fabric composite; Lubricant release; Optical self-reporting; Microcapsules; Tribofilm; Wear reduction

1. Introduction

Lubricant delivery and transfer-film formation govern friction stability and wear resistance in enclosed sliding contacts. Self-lubricating fabric composites are commonly used in aerospace actuators, heavy-duty bearings, articulated joints, and transmission systems. Their low density, mechanical compliance, and ability to operate without external lubrication make them suitable for enclosed sliding contacts [1–4]. During service, the sliding interface is buried between the fabric composite and the metallic counterpart, making lubricant release, redistribution, and transfer-film formation difficult to observe directly. This hidden nature of the contact interface limits the understanding of how lubricant supply evolves during sliding and how it contributes to interfacial protection [5, 6]. This makes it desirable to develop fabric composites that can not only protect the sliding interface but also provide an optical indication of lubricant release and accumulation.

Microencapsulation is an effective route for storing lubricants and releasing them under mechanical stress [7–10]. When embedded in polymer or fabric composites, oil-loaded microcapsules can rupture during sliding and deliver lubricant into the contact region, thereby reducing direct asperity contact and delaying lubrication failure [3, 4, 8]. However, most oil-loaded microcapsule systems are optically silent. Friction coefficient and wear volume describe the final tribological response, but they do not reveal when capsule rupture occurs, whether lubricant accumulates in the wear track, or how lubricant release is coupled with transfer-film formation. By contrast, the self-reporting polymers and mechanoresponsive microcapsules reported [11–14], primarily use optical changes to indicate mechanical damage, crack formation, healing, or other material-state changes. In these systems, the reporting component

is generally not the lubricant payload, and the optical signal is therefore not directly associated with lubricant delivery or tribofilm formation. An integrated microcapsule capable of coupling mechanically triggered lubricant release with an intrinsic optical readout is therefore still needed for self-lubricating fabric composites [15, 16].

Carbon quantum dots (CQDs) provide a promising route to bridge this functional gap because their intrinsic photoluminescence, nanoscale dimensions, and tunable surface chemistry allow an optical reporter to be incorporated directly into an oil-based lubricant reservoir [14, 17–19]. When dispersed in the lubricant phase, CQDs can therefore couple lubricant delivery with fluorescence-based tracking of the released phase. They may also provide tribological benefits because carbon-based nanomaterials can enter contact interfaces, reduce interfacial shear, and participate in transfer-film formation [20–25]. Nevertheless, pristine CQDs usually contain abundant polar groups and show poor compatibility with nonpolar lubricants such as polyalphaolefin (PAO) oils [17, 19, 23]. Aggregation or phase separation in oil may weaken fluorescence stability and limit their ability to indicate lubricant release and contribute to interfacial lubrication. Surface modification is therefore required to improve their dispersion in oil-based lubricant reservoirs while preserving their photoluminescence [23, 26].

In this work, dodecyl-chain-modified lipophilic CQDs (CQDs- C_{12}) were synthesized by esterification and used as fluorescent oil-compatible additives in PAO4. The CQDs- C_{12} /PAO4 phase was encapsulated in SiO_2 -based microcapsules and introduced into AF/PTFE fabric composites as lubricant reservoirs. Unlike conventional damage-reporting systems, the fluorescence signal in the present design originates from the same CQDs- C_{12} /PAO4 phase that

is delivered to the sliding interface, thereby directly linking the optical response to lubricant release and accumulation. The concentration of CQDs-C₁₂ in PAO4 was optimized by balancing fluorescence stability, oil-phase compatibility, and tribological performance.

2. Experiments

2.1. Materials

Citric acid (CA), L-cysteine (L-Cys), lauryl alcohol, p-toluenesulfonic acid monohydrate (PTSA), sodium hydroxide, ethanol, acetone, ethyl acetate, toluene, dichloromethane, petroleum ether, and n-octanol were purchased from Sinopharm Chemical Reagent Co., Ltd. Tetraethyl orthosilicate (TEOS) and cetyltrimethylammonium bromide (CTAB) were obtained from Shanghai Macklin Biochemical Co., Ltd. Polyalphaolefin oil 4 (PAO4) was used as the lubricant phase. Aramid fibers were purchased from Tayho Advanced Materials Co., Ltd. PTFE fibers were prepared in the laboratory. Phenolic acetal resin was supplied by Shanghai Xinguang Chemical Co., Ltd. The AF/PTFE hybrid fabric had a twill-weave architecture and an AF-to-PTFE mass ratio of 1:1. Its nominal warp and weft densities were 23.2 ends cm⁻¹ and 21.0 picks cm⁻¹, respectively, and its areal density was approximately 281 g m⁻². The nominal diameters of the AF and PTFE filaments were approximately 12 and 21 μm, respectively. AISI 1045 steel plates were used as supporting substrates, and AISI 1045 steel plates were used as supporting substrates.

2.2. Synthesis of CQDs and CQDs-C₁₂

Hydrophilic CQDs were synthesized by hydrothermal carbonization. CA (3.0 g) and L-Cys (1.5 g) were dissolved in 75 mL of ultrapure water, transferred into a 250 mL Teflon-lined stainless-steel autoclave, and heated at 180 °C for 24 h. After cooling, the solution was filtered

through a 0.22 μm membrane, dialyzed against ultrapure water using a 500 Da molecular-weight-cutoff dialysis bag for 24 h, and lyophilized to obtain CQDs.

Lipophilic CQDs-C₁₂ were prepared by acid-catalyzed esterification. CQDs (100 mg), lauryl alcohol (2.5 g), and PTSA (8 mg) were dispersed in 30 mL of toluene. The mixture was refluxed at 110 °C for 6 h using a Dean-Stark apparatus to remove generated water. After cooling, the crude product was extracted with water/dichloromethane (1:1, v/v). The organic phase was concentrated and purified by silica gel column chromatography to obtain CQDs-C₁₂.

2.3. Preparation of CD - MCs

CQDs-C₁₂/PAO4-loaded SiO₂ microcapsules (CD-MCs) were prepared by an oil-in-water emulsion-templated in situ polymerization process. CTAB (0.37 g) was dissolved in 100 mL of a water/ethanol continuous phase (90/10, v/v) at 50 °C. CQDs-C₁₂/PAO4 (1.26 g) was then added as the oil phase, corresponding to an oil-phase-to-water mass/volume ratio of 1.26 g: 90 mL and an oil-phase-to-total-continuous-phase ratio of 1.26 g:100 mL. The mixture was homogenized at 12,000 r/min for 5 min and subsequently ultrasonicated for 15 min. One drop of n-octanol was added as a defoaming agent, and the pH was adjusted to 10–11 using a 5 wt% NaOH aqueous solution. The emulsion was heated to 80 °C and stirred at 300 r/min while TEOS (1.2 g) was introduced using a syringe pump at 0.25 mL min⁻¹. After TEOS addition, the reaction was maintained at 80 °C and stirred at 600 r/min for 2 h.

2.4. Synthesis of fabric composites

The AF/PTFE twill fabric was ultrasonically cleaned with petroleum ether and acetone for 4 h, boiled in deionized water for 1 h, rinsed, and dried at 60 °C for 3 h. Phenolic resin (10 g)

was diluted with 70 mL of acetone/ethyl acetate/anhydrous ethanol (1:1:1, v/v/v). For SMCs/Fabric, Oil-MCs/Fabric, and CD-MCs/Fabric, the corresponding microcapsules were added at 5 wt% relative to the dry phenolic resin, equivalent to 0.50 g of microcapsules per 10 g of resin, and dispersed using a high-pressure cell disruptor. The pretreated fabric was impregnated with the resulting resin slurry and dried at 60 °C. The impregnation–drying cycle was repeated until the total matrix content reached 25–35 wt%. Accordingly, the microcapsules accounted for approximately 1.2–1.7 wt% of the final fabric composite, depending on the measured matrix uptake. Unless otherwise stated, all microcapsule-containing fabric composites contained 5 wt% microcapsules relative to dry phenolic resin. For CD-MCs/Fabric, the optimized core phase contained 5 wt% CQDs-C₁₂ relative to PAO4. The prepregs were then cut, bonded onto AISI 1045 steel substrates with phenolic resin adhesive, and hot-pressed at 140 °C and 0.2 MPa for 180 min.

The microcapsule-free AF/PTFE/phenolic resin composite was denoted as Pure Fabric. Composites containing empty SiO₂ microcapsules, PAO4-loaded microcapsules, and CQDs–C₁₂/PAO4-loaded microcapsules were denoted as SMCs/Fabric, Oil-MCs/Fabric, and CD-MCs/Fabric, respectively. Unless otherwise specified, “Fabric” refers to the cured AF/PTFE/phenolic resin fabric composite. Two additional control composites were prepared. CQDs/Fabric was prepared by directly dispersing pristine hydrophilic CQDs into the phenolic-resin slurry at the same nominal CQD mass as that introduced through the CD-MCs in CD-MCs/Fabric. CD-Oil/Fabric was prepared by directly blending the CQDs–C₁₂/PAO4 lubricant phase into the phenolic-resin slurry at the same nominal lubricant-phase mass as that introduced through the CD-MCs, but without SiO₂ encapsulation. All other impregnation, drying, hot-

pressing, and curing procedures were unchanged. Representative commercial products and peer-reviewed CQD and microcapsule-based lubrication systems are therefore summarized in **Table S3-S4**.

2.5. Characterization

Morphologies and microstructures were characterized by field-emission scanning electron microscopy (FE-SEM, JEOL JSM-7800F, Japan) and transmission electron microscopy (TEM, Thermo Scientific Talos F200X, USA). Cross-sectional transfer-film specimens were prepared using a focused ion beam system (FIB, Thermo Scientific Helios G4 UX, USA). **The tribofilm thickness was measured from the cross-sectional TEM image using ImageJ. The distance between the upper and lower tribofilm interfaces was measured perpendicular to the film at five positions distributed across the observable cross-sectional region, and the result is reported as the mean $\pm$ standard deviation.** Particle-size distributions were measured by dynamic light scattering (DLS, Malvern Zetasizer Nano ZS, UK). Chemical structures and surface compositions were analyzed by Fourier transform infrared spectroscopy (FTIR, Thermo Scientific Nicolet iS50, USA), X-ray photoelectron spectroscopy (XPS, Thermo Scientific ESCALAB 250Xi, USA), and nuclear magnetic resonance spectroscopy (NMR, Bruker Avance III 600 MHz, Germany). **Elemental analysis was performed using a vario EL cube analyzer (Elementar, Germany). TG analysis was conducted using a TG 209 F3 Tarsus analyzer (NETZSCH, Germany) from 30 to 400 °C at 10 °C min⁻¹ under nitrogen. The lubricant loading (LC) and encapsulation efficiency (EE) of CD-MCs were provided in **Table S1**.** Photoluminescence spectra and quantum yields were recorded using a fluorescence spectrophotometer (Hitachi F – 7000, Japan), and fluorescence lifetimes were measured using

a time-correlated single-photon counting spectrometer (Edinburgh Instruments FLS980, UK). A 45# steel ball with a diameter of 10 mm slid against the fabric composite specimen bonded to an AISI 1045 steel substrate. Tests were conducted at room temperature under a normal load of 40 N, a rotational speed of 120 r/min, and a duration of 2 h. The relative sliding motion followed a circular wear track with a radius of 5 mm, corresponding to a linear sliding speed of 0.0628 m s⁻¹ [27–29]. The steel ball was constrained against rolling, and the contact therefore operated under pure-sliding conditions with a slip ratio of 100%. The initial nominal contact pressures were estimated using a conventional Hertzian sphere-on-flat model. The calculation used an elastic modulus/Poisson's ratio of 210 GPa/0.30 for the steel ball and equivalent values of 3.4 GPa/0.38 for the AF/PTFE fabric composite. The nominal mean contact pressure and maximum Hertzian pressure were approximately 112 and 168 MPa, respectively. The coefficient of friction (COF) was continuously recorded. Each condition was repeated five times. After testing, the wear depth of the fabric composites was measured using a digital depth gauge. The wear volume was calculated from the geometric parameters of the circular wear track according to Equation:

$$\Delta V = 2\pi R \left[r^2 \arccos\left(\frac{r-h}{r}\right) - (r-h)\sqrt{2rh-h^2} \right]$$

where ΔV is the wear volume, R is the radius of the rotating wear track, r is the radius of the steel ball, and h is the measured wear depth.

2.6. Density functional theory calculations

Density functional theory (DFT) calculations were performed using the Quickstep module of CP2K 9.1 to examine the energetic feasibility of local ordering of CQD-derived sp²-rich carbon domains under the contact-zone constraints relevant to sliding. A reduced C₄₈H₂₀O₄

active-domain model consisting of two compositionally identical $C_{48}H_{20}O_4$ carbon fragments was constructed to represent locally exposed sp^2 -rich regions after release of CQDs- C_{12} into the tribological contact. The calculations employed the PBE exchange-correlation functional with Grimme D3(BJ) dispersion correction. A tilted/non-parallel configuration and a parallel graphitic-like stacked configuration were used as representative pre- and post-ordering states, respectively. Static energy comparisons, normal-approach scans, a seven-point constrained ordering path, lateral registry scans, and projected density-of-states (PDOS) analyses were performed. Full model definitions, computational parameters, coordinate construction, and data-processing procedures are provided in the **Supporting Information**.

3. Results and discussion

3.1. Molecular design and structural verification of lipophilic CQDs- C_{12}

Fig. 1 illustrates the molecular design and hierarchical fabrication strategy of the optically self-reporting and self-lubricating fabric composite. Hydrophilic CQDs synthesized from CA are incompatible with nonpolar PAO4.

Dodecyl chains were grafted onto CQDs through esterification with lauryl alcohol, producing CQDs- C_{12} with improved oil affinity. CQDs- C_{12} were dispersed in PAO4, encapsulated in SiO_2 -based microcapsules, and incorporated into AF/PTFE fabric composites by dipping and hot pressing.

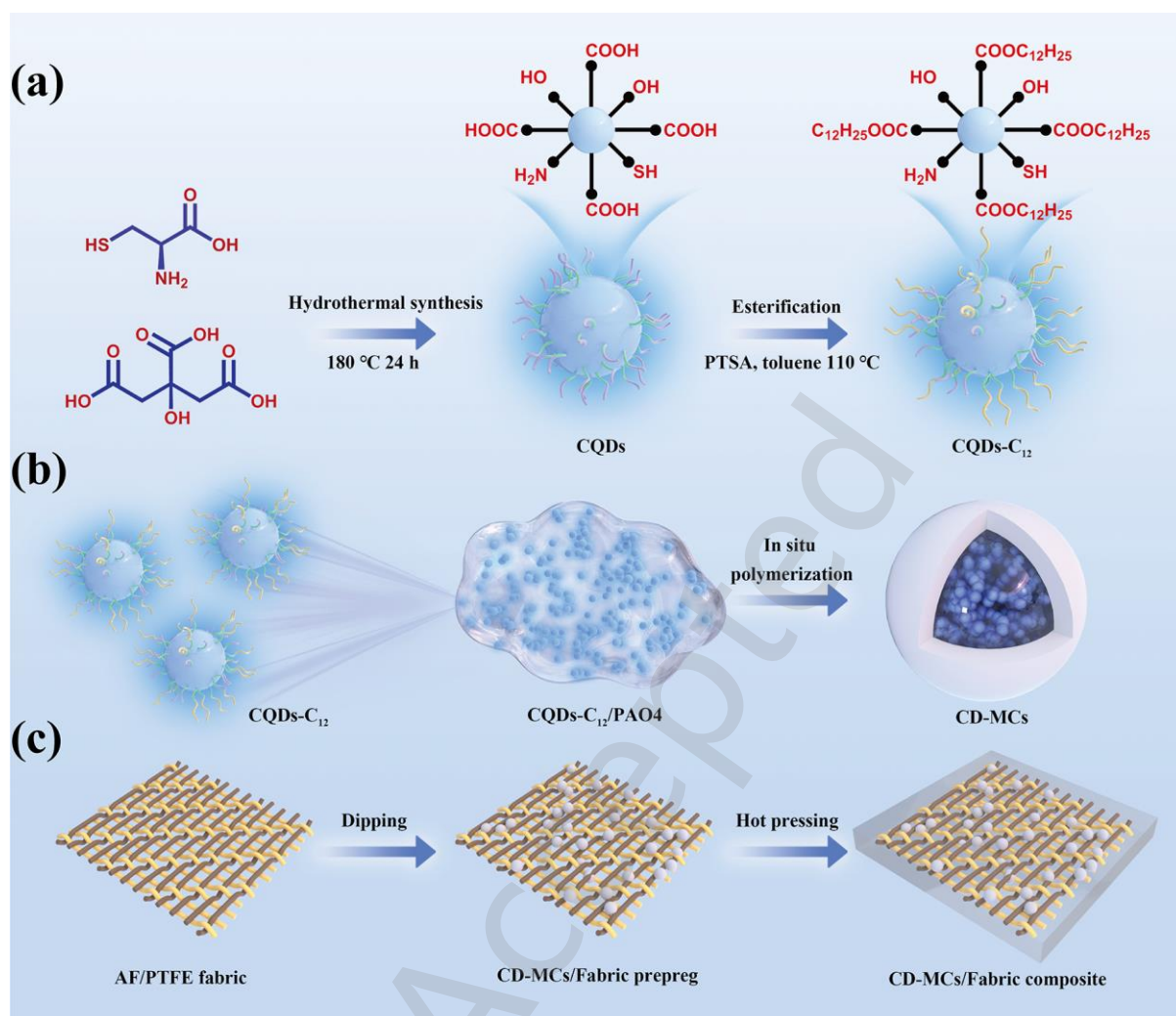


Fig. 1. Molecular design and fabrication of the optically self-reporting and self-lubricating fabric composite: (a) hydrothermal synthesis of CQDs and esterification to obtain lipophilic CQDs-C₁₂; (b) dispersion of CQDs-C₁₂ in PAO4 and encapsulation into SiO₂-based microcapsules; (c) fabrication of CD-MCs/Fabric by dipping and hot pressing.

The successful molecular grafting was confirmed by NMR spectroscopy, which revealed changes in the chemical environment. In the ¹H NMR spectrum (Fig. 2), pristine CDs showed signals at 5-7 ppm from aromatic protons on benzene-like domains, 0-3 ppm from saturated aliphatic protons, and 4-5 ppm from protons attached to C-O-linked carbon atoms. After modification, the proton signals shifted upfield, indicating that esterification changed the hydrogen environment near carboxyl groups. In addition, the strong signal at 1.4 ppm

confirmed the presence of aliphatic chains introduced by esterification, which became the dominant proton species in modified CDs.

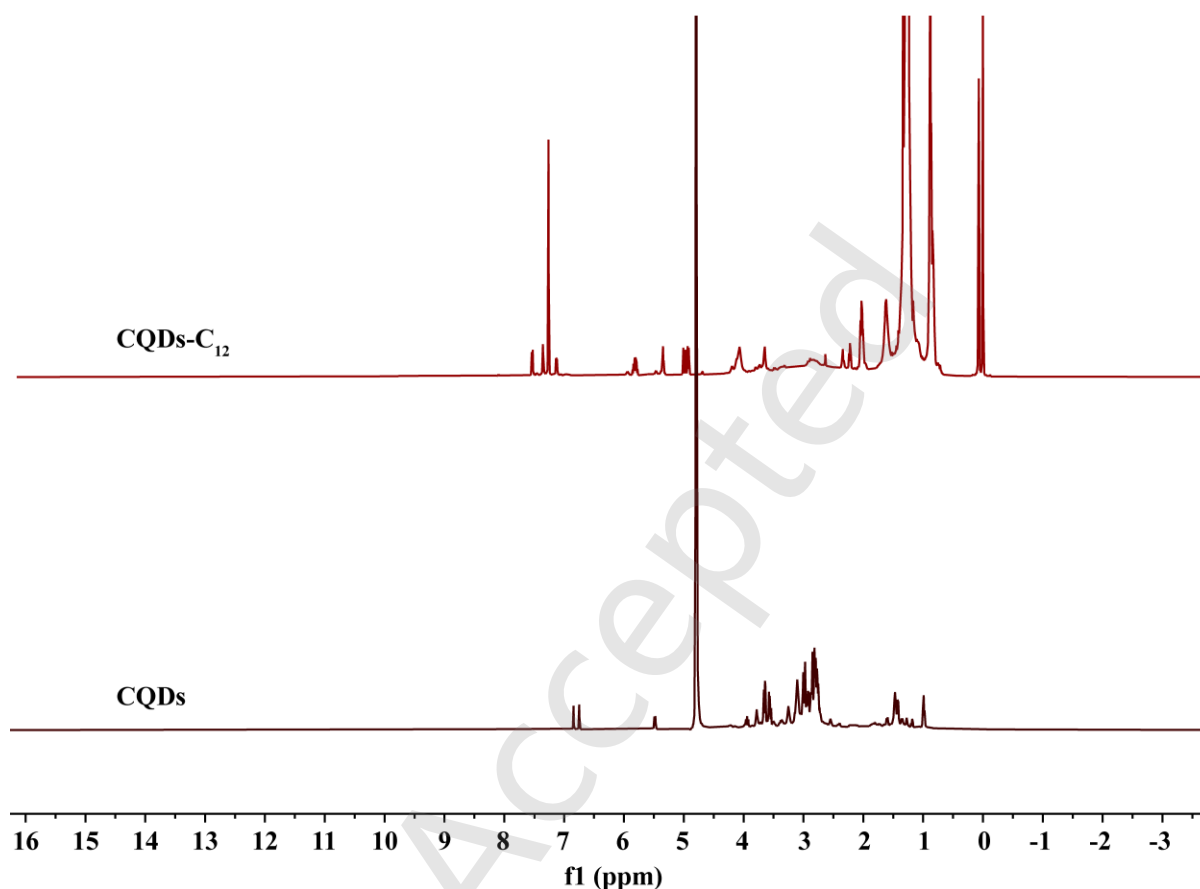


Fig. 2. ^1H NMR spectra of pristine CQDs and lipophilic CQDs-C₁₂. CQDs were measured in D_2O , while CQDs-C₁₂ were measured in CDCl_3 .

The ^{13}C NMR spectrum further verified the successful modification (Fig. 3). The carbonyl carbon signal of pristine CDs at 176 ppm shifted to 140 ppm in CDs-C₁₂, while the adjacent carbon peaks shifted to 110 ppm. New resonances also appeared in the 20-40 ppm region, which were attributed to the introduced aliphatic chains.

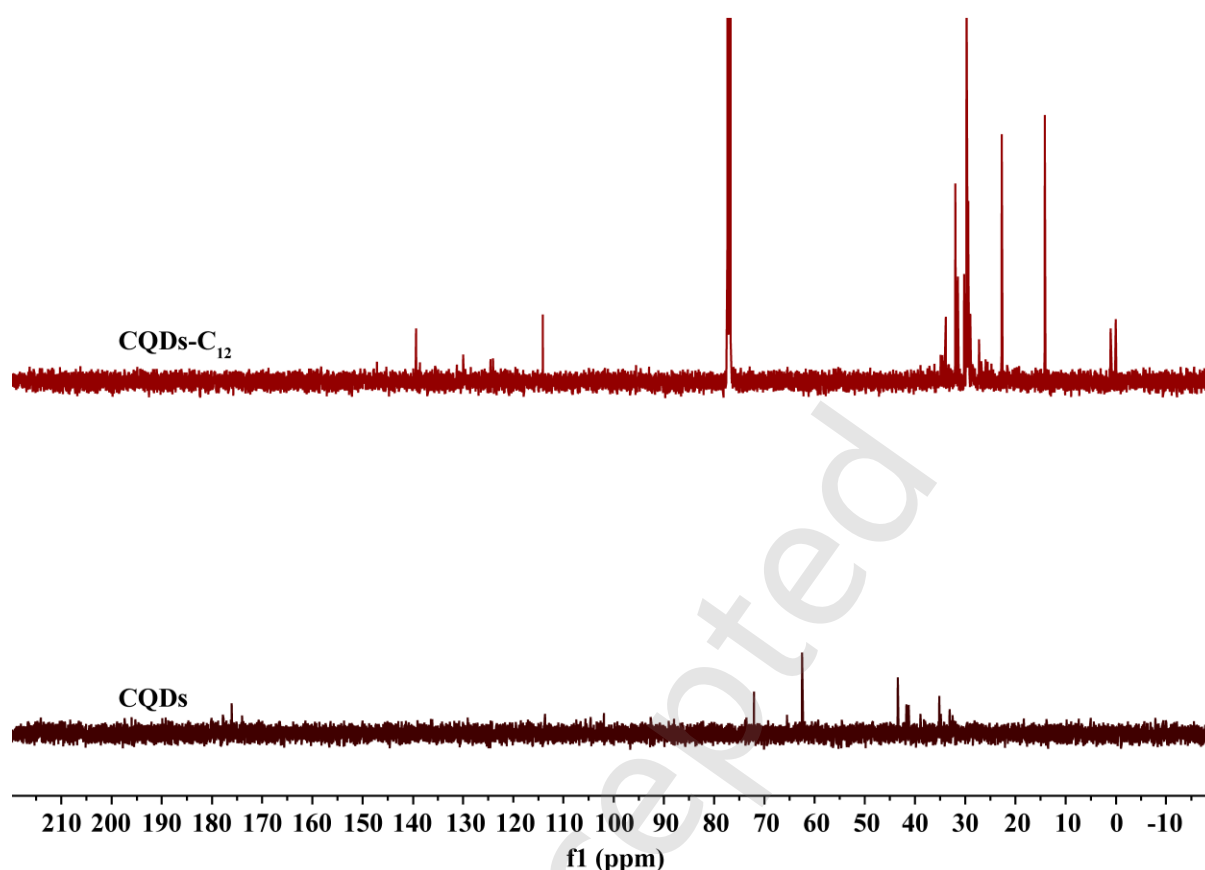


Fig. 3. ^{13}C NMR spectra of pristine CQDs and lipophilic CQDs- C_{12} . CQDs were measured in D_2O , while CQDs- C_{12} were measured in CDCl_3 .

The morphological and chemical changes induced by dodecyl-chain grafting were characterized by TEM, HRTEM, FTIR, and XPS (Fig. 4). Pristine CQDs display discrete quasi-spherical morphology with an average diameter of 2.90 ± 0.56 nm (Fig. 4a, b). After esterification with lauryl alcohol, CQDs- C_{12} formed spindle-shaped morphology with an axial length of 15.73 ± 5.26 nm and a radial width of 2.51 ± 0.51 nm (Fig. 4c-e). The radial width remained close to the size of pristine CQDs, whereas the axial length increased markedly, indicating that the nanoscale carbonaceous cores were retained but their interparticle packing was altered by the grafted alkyl chains.

HRTEM images and FFT patterns further revealed changes in local ordering (Fig. 4f, g). Pristine CQDs showed lattice spacings of 0.177 and 0.193 nm, indexed to the (104) and (012)

planes, respectively. CQDs-C₁₂ exhibited spacings of 0.296 and 0.298 nm, assigned to the (110) and ($\bar{1}\bar{1}0$) planes. These results suggest that dodecyl-chain grafting altered the interparticle packing and local ordering of CQDs-C₁₂, likely through alkyl-chain-mediated van der Waals interactions.

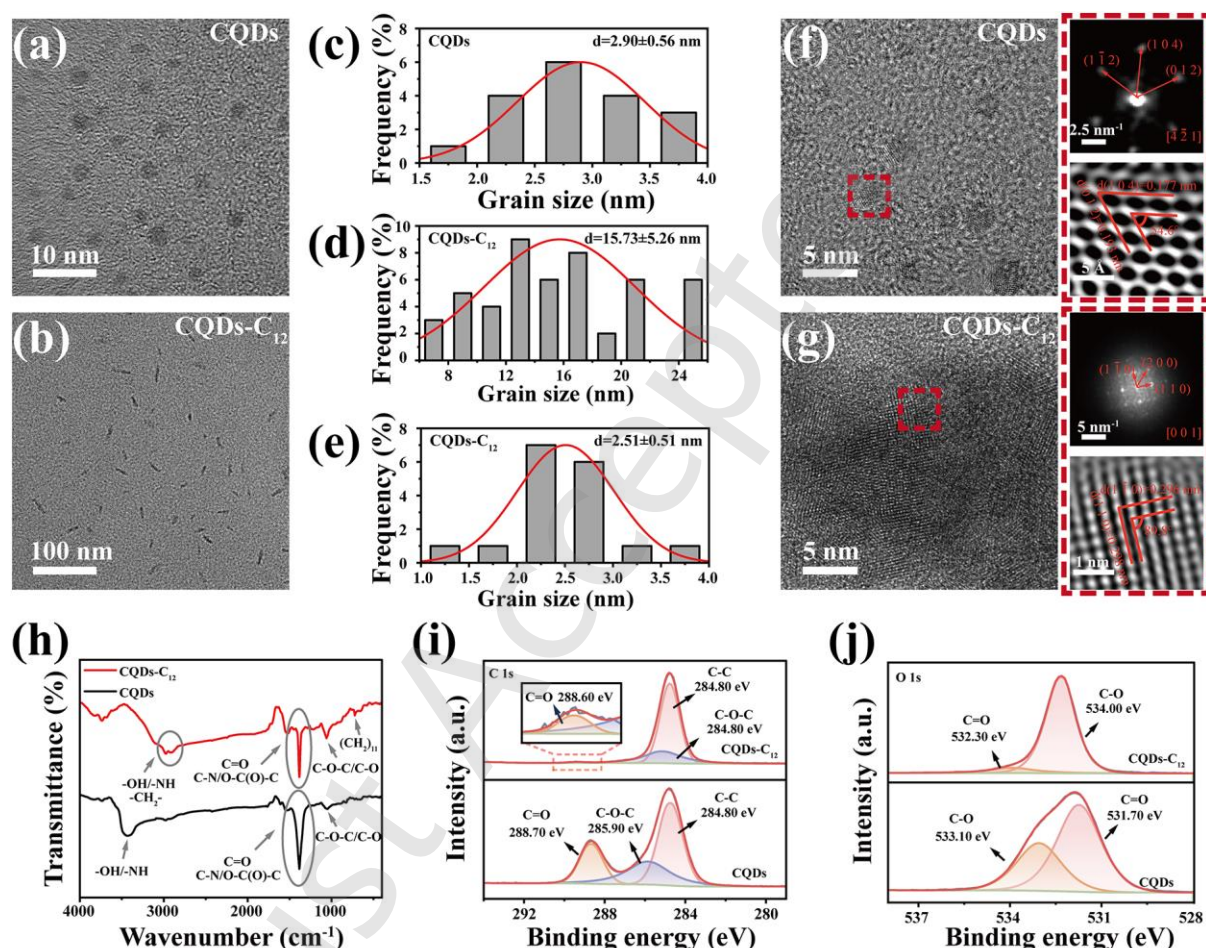


Fig. 4. Morphology and chemical characterization of CQDs and CQDs-C₁₂: TEM images of (a) CQDs and (c) CQDs-C₁₂; size distributions of (b) CQDs and (d, e) CQDs-C₁₂; HRTEM images and FFT patterns of (f) CQDs and (g) CQDs-C₁₂; (h) FTIR spectra; high-resolution XPS spectra of (i) C 1s and (j) O 1s.

FTIR spectra confirmed the functional group change after modification (Fig. 4h). The broad -OH/-NH band around 3400 cm⁻¹ in pristine CQDs weakened after esterification. The

carbonyl-related band near 1620 cm^{-1} became more pronounced. Additional CQDs-C₁₂ bands at 2923 and 723 cm^{-1} corresponded to $-\text{CH}_2-$ stretching and rocking vibrations of long alkyl chains, respectively, supporting the successful introduction of dodecyl chains onto the CQDs surface.

The bulk elemental composition and thermal behavior of CQDs-C₁₂ were further evaluated by elemental analysis and TG, respectively. As summarized in **Table S2**, the C, H, N, S, and O contents were 61.5, 3.8, 12.4, 1.1, and 21.2 wt%, respectively. The carbon- and hydrogen-containing composition is consistent with a carbonaceous framework bearing alkyl-containing surface species, while the detectable N, S, and O contents indicate the retention of heteroatom-containing and oxygenated surface functionalities.

As shown in **Fig. S3**, CQDs-C₁₂ exhibited a small and gradual mass loss below approximately $150\text{ }^{\circ}\text{C}$, followed by a more pronounced mass-loss stage between approximately 150 and $350\text{ }^{\circ}\text{C}$. These changes can be attributed to the removal of adsorbed volatile species and the subsequent decomposition of surface organic functionalities, including alkyl-containing species. After normalization of the initial mass, the residual mass at $400\text{ }^{\circ}\text{C}$ was approximately 83.3 wt%, corresponding to a total mass loss of approximately 16.7 wt%. The relatively high residual fraction indicates that a thermally stable carbonaceous framework remained after decomposition of the surface organic components.

XPS analysis provided further evidence for the surface chemical changes caused by dodecyl-chain grafting. The survey spectra showed C, O, N, and S signals for pristine CQDs, whereas CQDs-C₁₂ mainly showed C and O signals (Fig. 5). Quantitative analysis revealed that the carbon content increased from 57.90% to 71.68%, while the oxygen content decreased

from 33.88% to 28.31% (Table 1). The N and S signals, detected in pristine CQDs at 3.22% and 4.92%, respectively, became undetectable after modification. The N and S signals became undetectable in CQDs-C₁₂, suggesting effective surface shielding by the grafted alkyl chains rather than complete removal of heteroatom-containing domains from the carbon core.

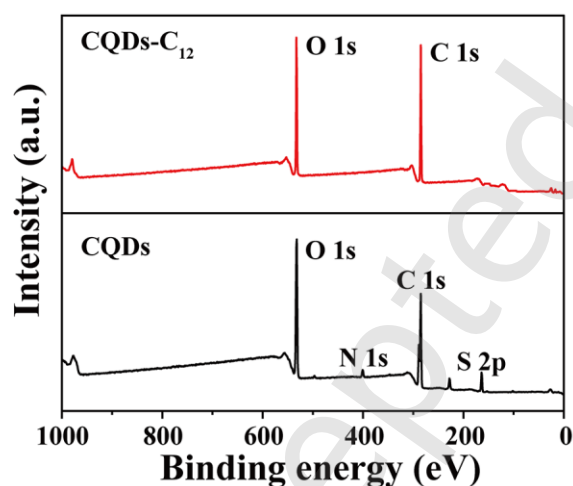


Fig. 5. XPS survey spectra of CQDs and CQDs-C₁₂.

Table 1. Elemental compositions of CQDs and CQDs-C₁₂ determined from XPS survey spectra.

Samples	C	O	N	S
CQDs	57.90%	33.88%	3.22%	4.92%
CQDs-C ₁₂	71.68%	28.31%	—	—

High-resolution C 1s and O 1s spectra further clarified the surface composition changes (Fig. 4i, j; Tables 2 and 3). In the C 1s spectrum, the C-C/C=C component increased from 28.59% to 53.26% after modification, consistent with the incorporation of hydrocarbon-rich dodecyl chains. The C-O-C/C-O component changed only slightly, whereas the C=O component decreased markedly, suggesting that part of the original carboxyl-containing

surface groups was consumed or shielded during esterification. In the O 1s spectrum, the C–O contribution decreased from 11.98% to 2.68%, while the C=O-related contribution increased from 21.68% to 25.63%, indicating a redistribution of oxygen-containing surface species after esterification.

Table 2. Deconvoluted C 1s components of CQDs and CQDs–C₁₂.

C 1s	C–C (284.80 eV)	C–O–C (285.90 eV)	C=O (288.70 eV)
CQDs	28.59%	16.11%	13.20%
CQDs–C ₁₂	53.26%	17.82%	0.60%

Table 3. Deconvoluted O 1s components of CQDs and CQDs–C₁₂.

O 1s	C–O (533.10 eV)	C=O (531.70 eV)
CQDs	11.98%	21.68%
CQDs–C ₁₂	2.68%	25.63%

High-resolution N 1s and S 2p spectra also supported the surface-shielding effect (Fig. 6). Pristine CQDs showed N 1s components assigned to C–N and C–N–H species, together with S 2p signals from sulfur-containing groups. In contrast, these signals became undetectable for CQDs–C₁₂, consistent with the survey spectra. Overall, the TEM, HRTEM, FTIR, and XPS results demonstrate that dodecyl chains were successfully grafted onto CQDs. This modification reduced the exposure of polar groups and increased the hydrocarbon character of the particle surface, thereby improving the compatibility of CQDs–C₁₂ with nonpolar PAO4.

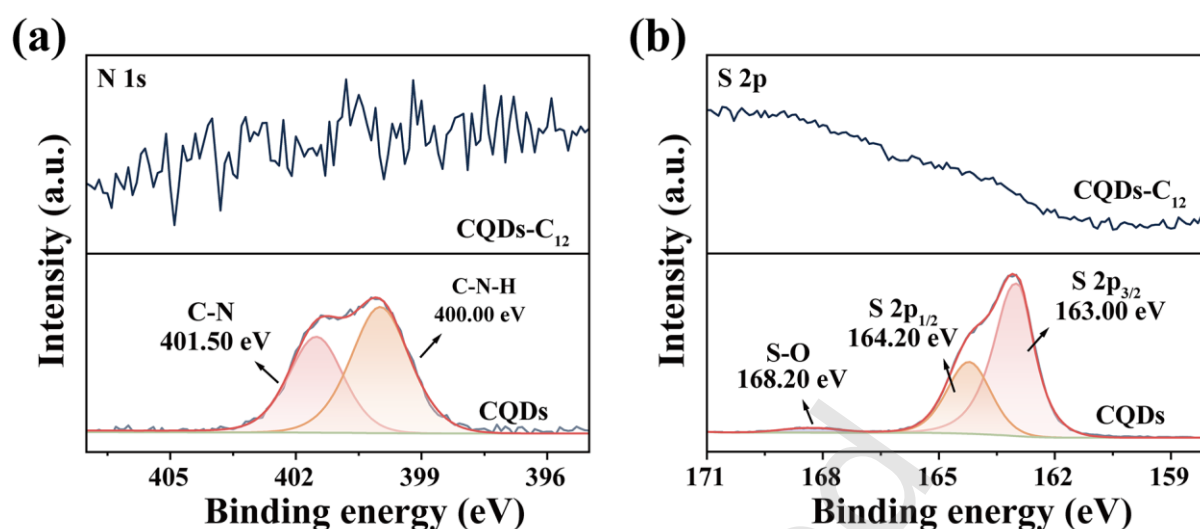


Fig. 6. High-resolution XPS spectra of heteroatom-related regions. (a) N 1s and (b) S 2p spectra of CQDs and CQDs-C₁₂.

3.2. Optical and PAO4-phase stability of CQDs-C₁₂ for self-reporting lubrication

The optical properties of CQDs and CQDs-C₁₂ were tested to determine whether dodecyl-chain grafting preserved the fluorescence response required for release reporting (Fig. 7). The time-resolved photoluminescence (TRPL) decay curves showed similar profiles for CQDs and CQDs-C₁₂, nearly identical average lifetimes of 8.93 and 8.89 ns, respectively (Fig. 7a, b), indicating that esterification did not significantly disturb the intrinsic emissive centers of CQDs. Steady-state photoluminescence spectra also showed only slight shifts in the excitation/emission maxima, from 369/433 nm for CQDs to 370/436 nm for CQDs-C₁₂ (Fig. 7c). Under UV irradiation, both samples emitted blue fluorescence, confirming that the optical activity of CQDs was retained after dodecyl-chain modification.

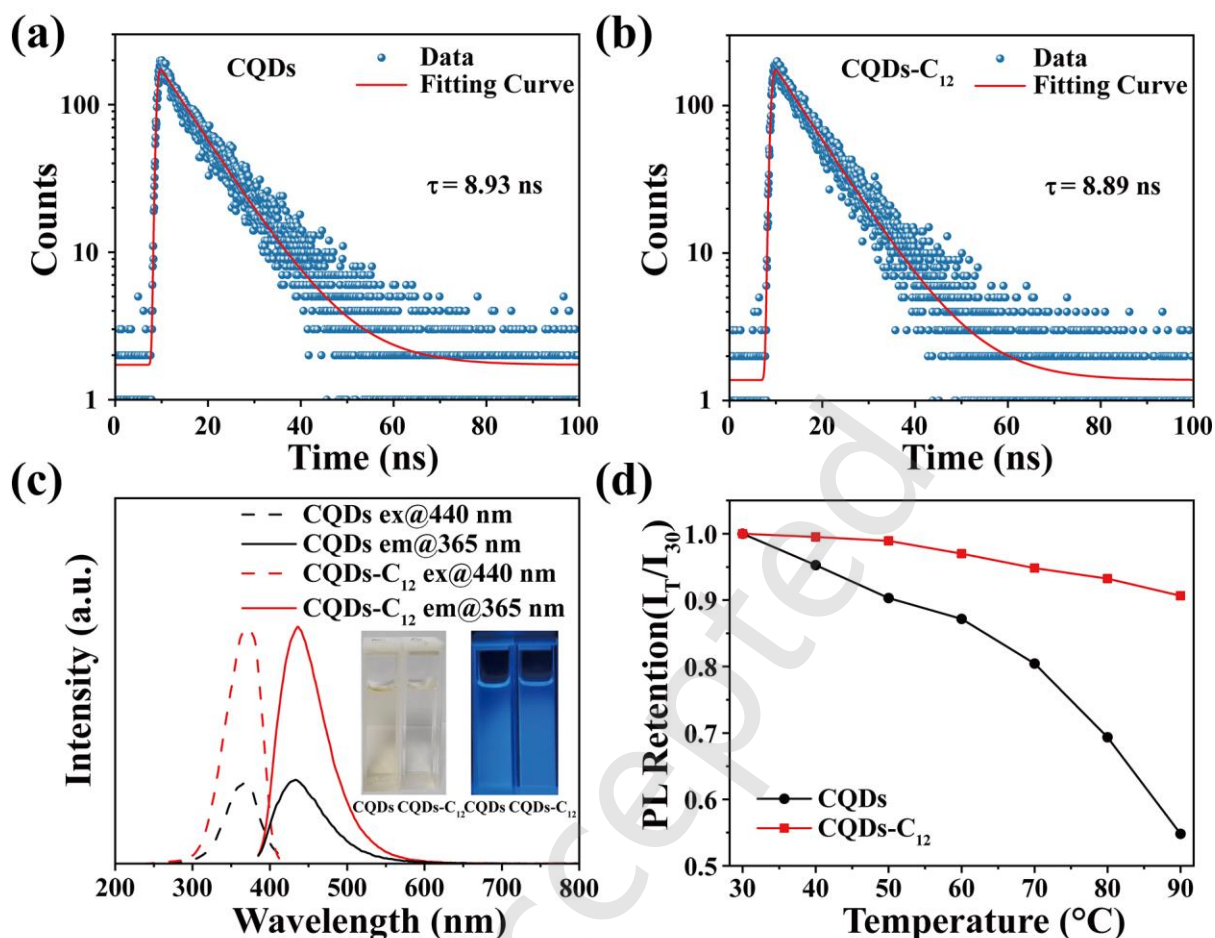


Fig. 7. Optical properties and thermal stability of CQDs and CQDs-C₁₂: TRPL decay curves of (a) CQDs and (b) CQDs-C₁₂; (c) excitation and emission spectra, with photographs under visible light and UV irradiation in the inset; (d) temperature-dependent photoluminescence retention.

The thermal stability of the fluorescence signal was tested because local temperature fluctuations may occur during sliding. As shown in Fig. 7d, pristine CQDs retained approximately half of their initial PL intensity at 90 °C, whereas CQDs-C₁₂ retained 90.66%. This improved resistance to thermal quenching can be attributed to the hydrophobic dodecyl-chain shell, which can restrict the thermal motion of surface emissive groups and suppress non-radiative relaxation. The high PL retention suggests that CQDs-C₁₂ can maintain a detectable fluorescence signal under thermally fluctuating tribological conditions.

The long-term compatibility of CQDs-C₁₂ with PAO4 was tested over 32 weeks at a concentration of 5 mg mL⁻¹ by optical observation, DLS, and PL retention measurements (Fig. 8 and Fig. 9). The CQDs-C₁₂/PAO4 dispersion maintained homogeneous blue fluorescence under UV irradiation, with no visible precipitation or phase separation during storage (Fig. 8a). DLS profiles showed gradual broadening of the apparent size distribution and the emergence of larger-size populations over time (Fig. 8b-j), indicating limited association or soft clustering of CQDs-C₁₂ in the oil phase. These associated structures remained suspended in the viscous PAO4 medium, and the dispersion remained macroscopically stable. The stored dispersion also retained more than 90% of its initial PL intensity after 32 weeks (Fig. 9), confirming its long-term optical stability. These results demonstrate that CQDs-C₁₂ retain their fluorescence after surface modification and exhibit sufficient thermal, optical, and dispersion stability in PAO4, supporting their use as oil-compatible fluorescent labels in PAO4-loaded microcapsules for self-reporting lubrication.

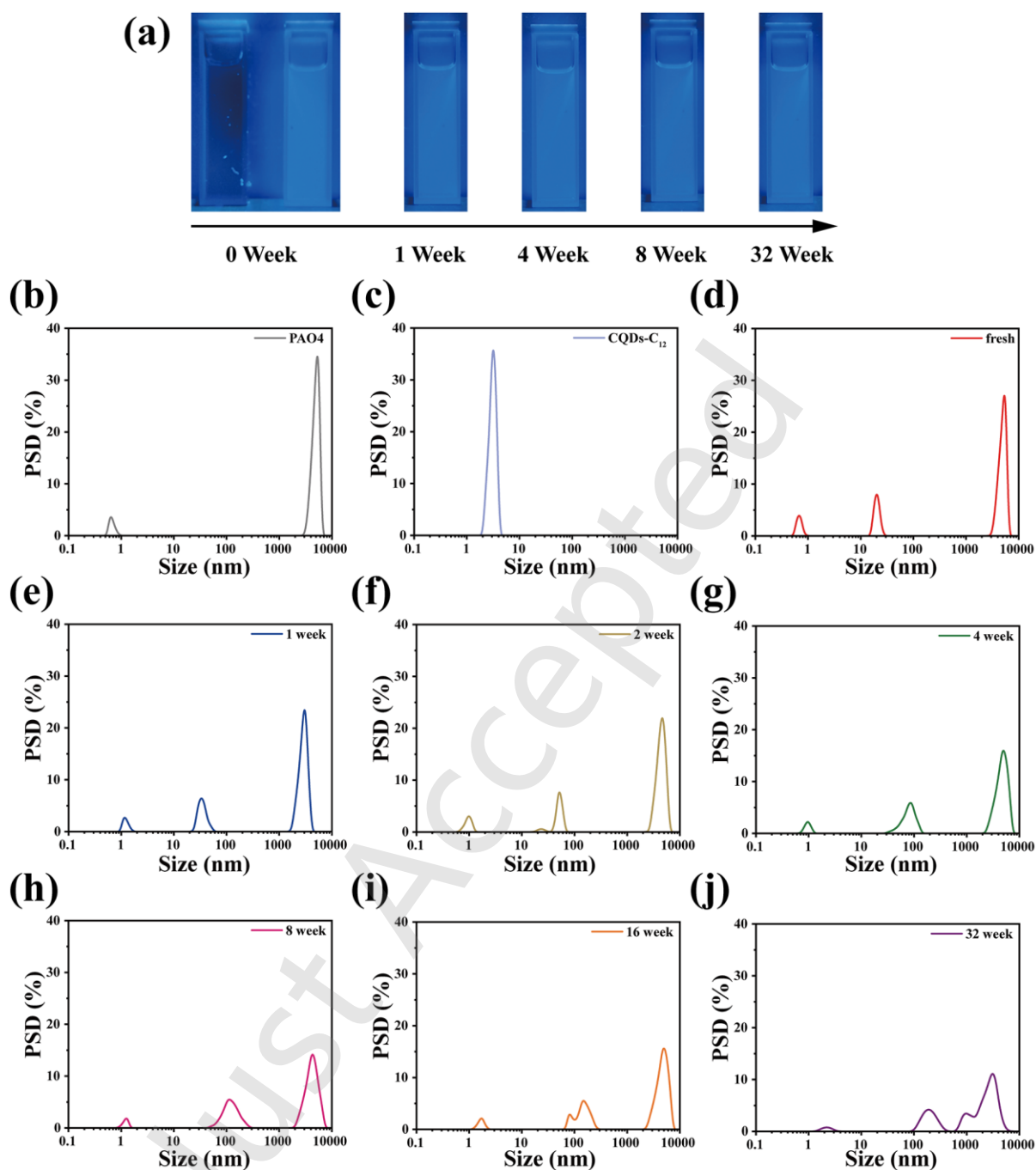


Fig. 8. Long-term dispersion stability of CQDs-C₁₂ in PAO4. (a) Photographs of CQDs-C₁₂/PAO4 dispersion under UV irradiation after storage for 0, 1, 4, 8, and 32 weeks. (b, c) DLS profiles of PAO4 and CQDs-C₁₂. (d-j) DLS profiles of CQDs-C₁₂/PAO4 after storage for 0, 1, 2, 4, 8, 16, and 32 weeks.

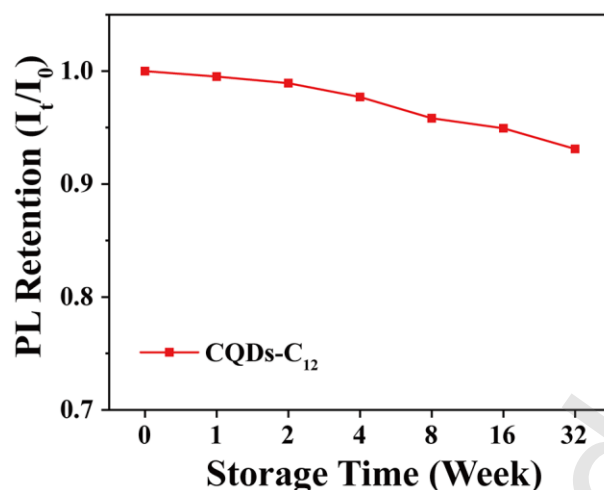


Fig. 9. Long-term optical stability of CQDs–C₁₂ in PAO4. PL retention of CQDs–C₁₂/PAO4 dispersion as a function of storage time over 32 weeks.

3.3. Fluorescence self-reporting of lubricant release and tribological performance

To integrate lubricant release with optical self-reporting, the CQDs–C₁₂/PAO4 lubricant phase was encapsulated into SiO₂-based microcapsules. SEM images show that the resulting CD-MCs exhibited a predominantly spherical morphology and a distinct shell-like structure (Fig. 10). Quantitative measurements from five preparation batches yielded an average lubricant loading of 67.2±4.4 wt% and an encapsulation efficiency of 64.1±2.3%. The corresponding encapsulated lubricant-phase mass was 0.807±0.029 g per batch. SEM examination of fractured microcapsules revealed local shell-wall thicknesses ranging from 48 to 87 nm, with an average value of 66.7±17.2 nm (Table S1 and Fig. S2). These quantitative results establish the core-loading capacity and nanoscale shell architecture of the CD-MCs. After incorporation into the AF/PTFE fabric composite, the capsules are expected to rupture under local contact stress during sliding and release PAO4 and CQDs–C₁₂ into the friction interface.

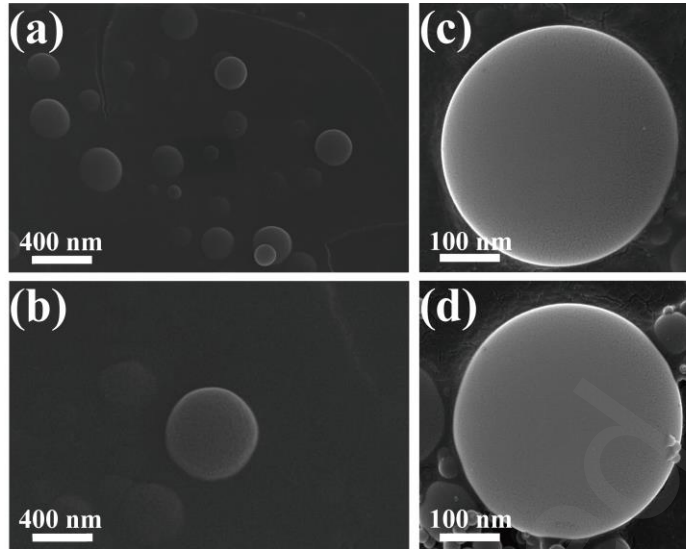


Fig. 10. Morphology of SiO₂-based microcapsules. SEM images showing spherical microcapsules with submicrometer-scale dimensions.

The concentration of CQDs–C₁₂ in PAO4 was optimized to balance fluorescence intensity and tribological performance (Fig. 11). As shown in Fig. 11a, increasing the concentration from 1% to 5% resulted in an enhancement of PL intensity, while the emission peak position and FWHM remained nearly unchanged. When the concentration was further increased to 10%, the emission maximum redshifted from 445 nm for the 5% sample to 456 nm, accompanied by FWHM broadening from 68 to 73 nm.

The tribological performance of fabric composites containing CD–MCs was tested (Fig. 11b-d). As the CQDs–C₁₂ concentration in PAO4 increased from 1% to 10%, the average friction coefficient decreased overall, while the wear volume remained low with slight variations. Although the 10% sample gave the lowest friction coefficient, it showed spectral redshift and broadening in the PL analysis. In contrast, the 5% formulation provided strong fluorescence, stable spectral features, and effective friction and wear reduction. **The formulation containing 5 wt% CQDs–C₁₂ in PAO4 was selected for the subsequent tests. To**

evaluate the adaptability of CD-MCs/Fabric to variations in load and extended sliding duration, additional 2 h tests were conducted at 20 and 60 N, together with a 6 h continuous sliding test at 40 N (**Fig. S9**). As shown in **Fig. S9a-b**, CD-MCs/Fabric exhibited relatively stable low-friction behavior after running-in at both 20 and 60 N. Together with the original 40 N result, this indicates that its friction-reducing performance was maintained over the investigated load range of 20-60 N. **Fig. S9c** further shows that a relatively stable frictional response was retained during 6 h of continuous sliding, indicating extended-duration tribological stability

To further quantify the time-dependent fluorescence response, the background-corrected emission spectra were integrated over 400–600 nm, and the resulting integrated areas were normalized to the value obtained after 2 h of sliding. As shown in **Fig. S1**, the normalized integrated fluorescence area increased monotonically with sliding time, reaching approximately 35.6%, 71.2%, 87.8%, and 100.0% after 0.5, 1.0, 1.5, and 2.0 h, respectively. Because the CQDs–C₁₂ concentration in PAO4 and the fluorescence-acquisition conditions were maintained constant, the normalized integrated fluorescence area can be used as a semi-quantitative indicator of the relative amount of fluorescent lubricant released and accumulated in the wear track. The fluorescence response increased rapidly during the first hour and subsequently increased at a lower rate, indicating an initial rapid-release stage followed by slower release and interfacial accumulation.

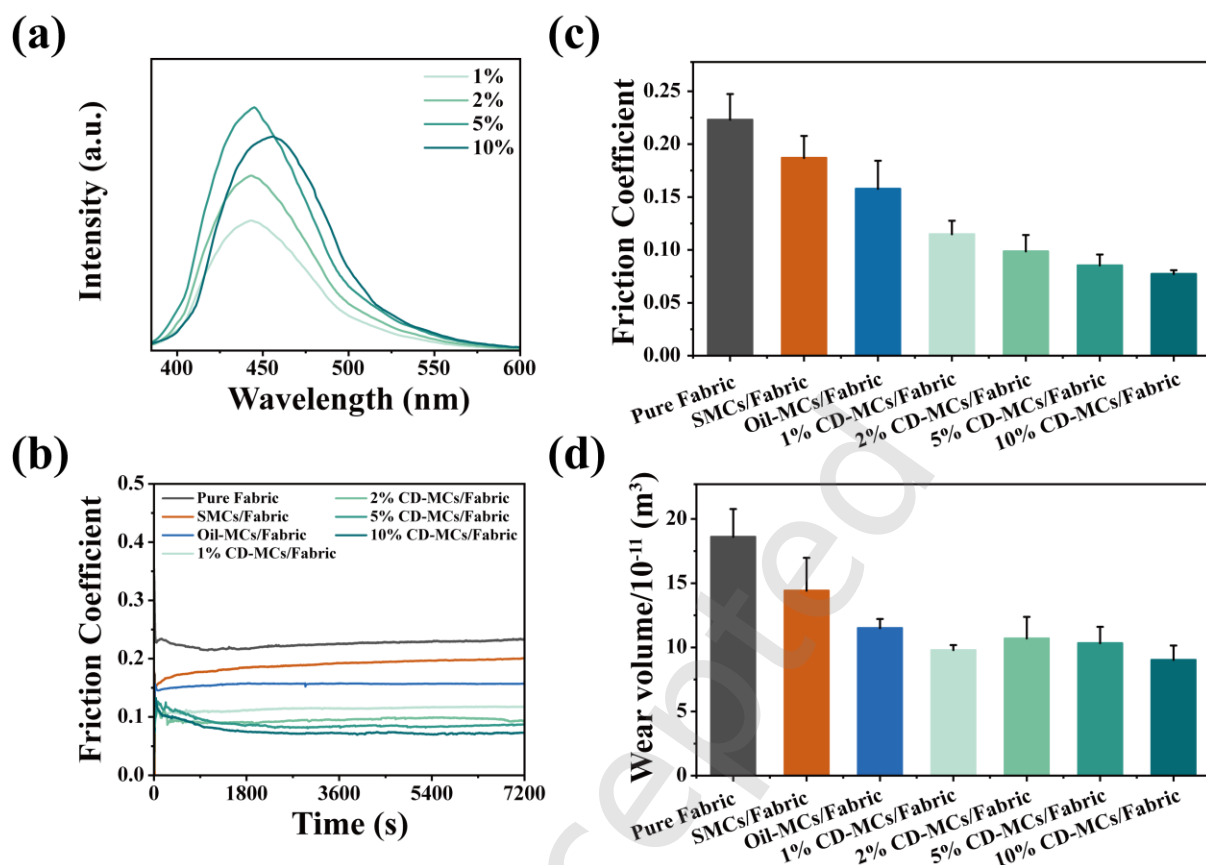


Fig. 11. Effects of the CQDs-C₁₂ concentration in the PAO4 core phase on the optical and tribological performance of CD-MCs/Fabric. (a) PL spectra of CQDs-C₁₂/PAO4 dispersions containing 1, 2, 5, and 10 wt% CQDs-C₁₂; (b) time-dependent friction-coefficient curves; (c) Mean friction coefficients and (d) wear volumes; error bars represent SD (n = 5). The values of 1, 2, 5, and 10 wt% denote the CQDs-C₁₂ concentration in the PAO4 core phase, not the microcapsule content in the fabric composite.

The lubricant-release behavior and tribological performance of CD-MCs/Fabric composites were tested (Fig. 12). Time-dependent fluorescence imaging visualized the progressive appearance and accumulation of the fluorescent lubricant phase in the wear track (Fig. 12a), while in situ fluorescence spectra showed a monotonic increase over 2 h (Fig. 12b), consistent with cumulative release and accumulation. A systematic bathochromic shift was observed, with the emission peak migrating from 443 nm to 452 nm and the FWHM broadening

393 from 69 to 75 nm.

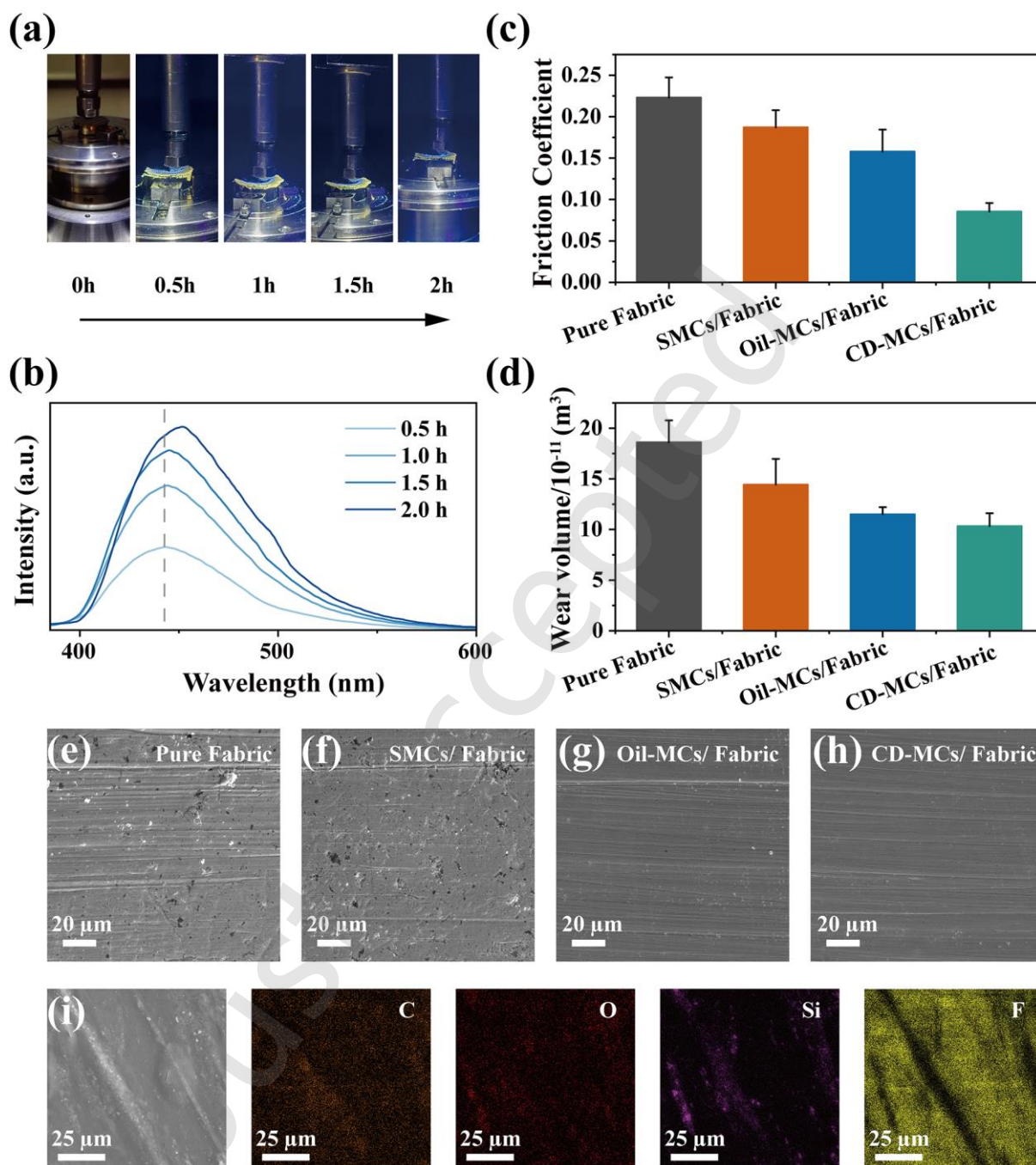


Fig. 12. Fluorescence self-reporting of lubricant release and tribological performance of fabric composites. (a) Time-dependent fluorescence images during sliding. (b) Fluorescence spectra collected at different sliding times. (c,d) Average friction coefficients and wear volumes. (e-h) SEM images of worn counterpart steel ball surfaces. (i) SEM image and EDS maps of C, O, Si, and F on the worn steel ball surface after sliding against CD-MCs/Fabric.

These changes suggest local enrichment of CQDs–C₁₂ and a changed microenvironment after release from microcapsules. Compared with Pure Fabric, SMCs/Fabric, and Oil–MCs/Fabric, CD–MCs/Fabric showed the lowest friction coefficient of about 0.085 and the lowest wear volume of about $1.03 \times 10^{-10} \text{ m}^3$ (Fig. 12c, d), confirming effective friction and wear reduction.

To further distinguish the roles of CQD surface modification and lubricant microencapsulation, representative time-dependent friction curves were obtained for two additional control composites (Fig. S6 and S7). For CQDs/Fabric, which was prepared by directly incorporating pristine hydrophilic CQDs into the phenolic matrix, the friction coefficient gradually increased from approximately 0.18 during running-in and subsequently approached approximately 0.20. This value was markedly higher than that of CD–MCs/Fabric (~0.085), indicating that direct addition of pristine hydrophilic CQDs did not reproduce the low-friction behavior of the oil-compatible microcapsule system. The microcapsule-free CD–Oil/Fabric control exhibited a rapid initial increase in friction coefficient, followed by a relatively stable value of approximately 0.19 during most of the 2 h test. Its friction coefficient remained substantially higher than that of CD–MCs/Fabric. These comparisons support that C₁₂ modification is important for integrating CQDs into the PAO4-based fluorescent lubricant reservoir, whereas SiO₂ microencapsulation helps regulate lubricant availability and maintain effective interfacial lubrication during prolonged sliding.

The worn counterpart steel ball surface also became much smoother, with mainly fine and shallow wear traces (Fig. 12e-h). EDS mapping further showed the coexistence of C, O, Si, and F in the wear track (Fig. 12i), indicating that released CQDs–C₁₂/PAO4, fragmented

SiO₂ shells, and PTFE/AF transfer components jointly contributed to the formation of a multi-component protective layer (Fig. 13).

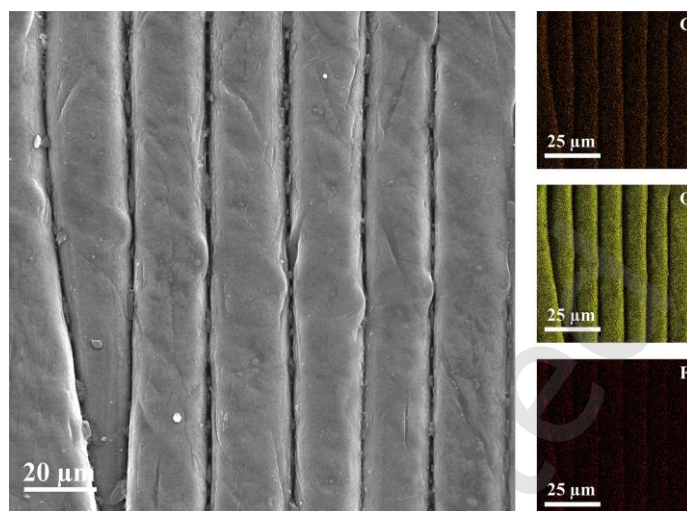


Fig. 13. Characterization of Pure Fabric. (a) SEM image. (b-d) EDS maps of C, O, and F.

SEM images of fabric composites worn surfaces were shown in Fig. 14. SMCs/Fabric showed a rough worn surface with visible abrasive marks and adhered wear debris, indicating limited protection from empty SiO₂ microcapsules. Oil-MCs/Fabric displayed a smoother surface with shallower scratches, suggesting that released PAO4 promoted milder boundary-lubricated sliding. CD-MCs/Fabric showed the smoothest worn surface among the three samples, with fewer debris and fine shallow scratches, confirming the enhanced lubrication effect of CQDs-C₁₂/PAO4-loaded microcapsules. EDS mapping of the CD-MCs/Fabric wear track showed Si and O signals across the worn region.

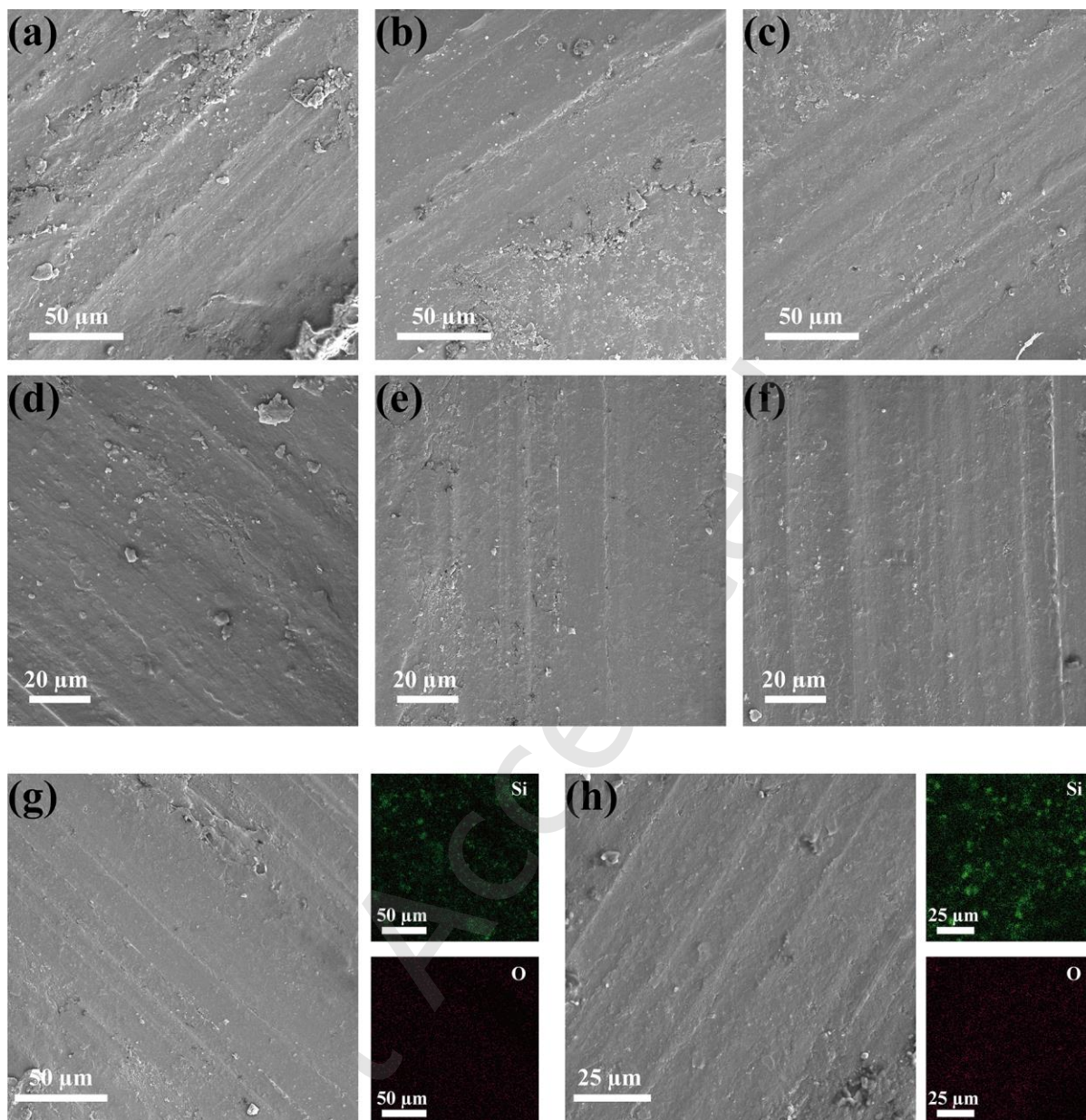


Fig. 14. Worn surface morphologies of fabric composites after ball-on-disk sliding. SEM images of (a, d) SMCs/Fabric, (b, e) Oil-MCs/Fabric, and (c, f) CD-MCs/Fabric. (g, h) SEM images and EDS maps of Si and O on worn CD-MCs/Fabric surfaces.

3.4. Formation of the continuous multi-phase tribofilm on the counterpart steel ball

Cross-sectional FIB-TEM was performed on the worn counterpart steel ball of CD-MCs/Fabric (Fig. 15a, b). TEM images show a compact and continuous tribofilm between the Pt layer and the steel substrate (Fig. 15c). The film is about 38.10 ± 0.1 nm thick and covers the

worn surface, indicating that released lubricant components and transferred species were incorporated into a continuous interfacial layer (Fig. S4) [30, 31].

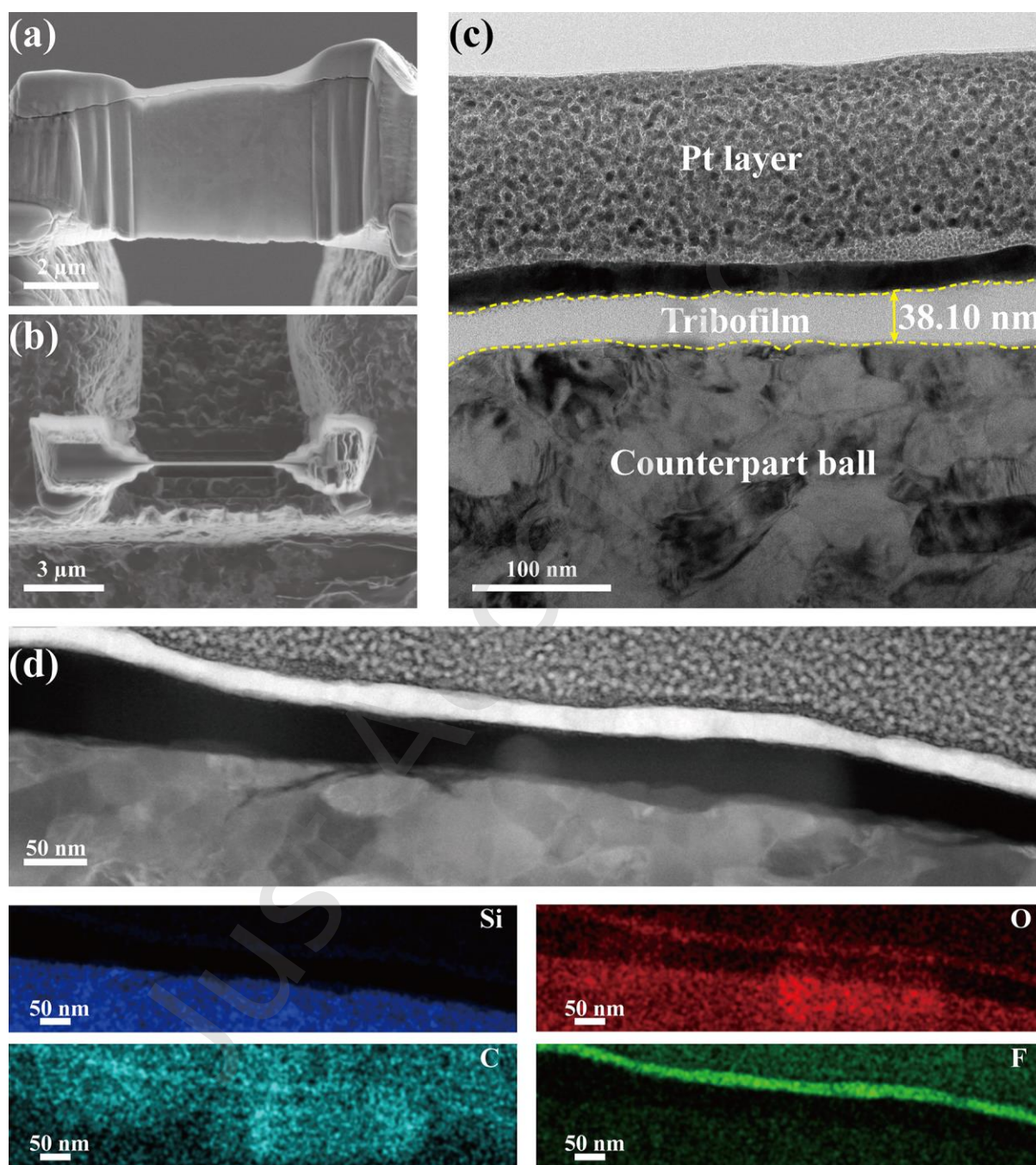


Fig. 15. Cross-sectional FIB-TEM characterization of the multicomponent tribofilm; (a,b) FIB lift-out images; (c) cross-sectional TEM image of the tribofilm; (d) EDS maps of Si, O, C, and F.

EDS maps confirm the multi-component composition of the tribofilm (Fig. 15d). C and O

are distributed within the interfacial layer, while Si and F are also detected. Si originates from fragmented SiO₂ shells, and F comes from PTFE transfer from the AF/PTFE fabric. The C signal is associated with CQDs–C₁₂-derived carbonaceous species, PAO4-derived species, and substrate residues. These results show that released CQDs–C₁₂/PAO4, silica fragments, and AF/PTFE fabric jointly form the protective tribofilm.

Raman spectroscopy was further employed to characterize the carbonaceous structures of the tribofilms formed on the counterpart steel balls (**Fig. S5**). The D band is associated with defect-activated vibrations of aromatic sp²-carbon domains, whereas the G band primarily originates from the in-plane stretching vibration of sp²-bonded carbon. The I_D/I_G ratios of the tribofilms formed by Pure Fabric, SMCs/Fabric, Oil-MCs/Fabric, and CD-MCs/Fabric were 0.48, 0.52, 0.63, and 1.54, respectively. Compared with the matched Oil-MCs/Fabric control, CD-MCs/Fabric exhibited a substantially enhanced defect-activated D-band contribution. This result indicates that the introduction of CQDs-C₁₂ was associated with the formation of a more defect-rich and edge-rich sp² carbonaceous structure in the tribofilm.

XPS was subsequently used to examine the chemical states of the tribofilm formed on the worn steel counterpart after sliding against CD-MCs/Fabric (**Fig. S8**). The survey spectrum confirmed the presence of C, O, Fe, and Si. The high-resolution C 1s spectrum was dominated by a C–C/C=C component at 284.80 eV, accompanied by C–O/C–O–C and C=O/O–C=O components at 286.27 and 288.80 eV, respectively. The corresponding O 1s spectrum contained Fe–O, C=O/Fe–OH, and Si–O–Si/C–O components centered at 530.66, 531.80, and 532.43 eV, respectively.

HRTEM and FFT analyses further revealed several locally ordered domains within the

tribofilm, with lattice spacings of 0.192–0.198, 0.241, and 0.341 nm (**Fig. 16**). The 0.341 nm spacing is consistent with the interlayer spacing of graphitic (002) planes. Together with the defect-rich sp^2 -carbon signatures revealed by Raman spectroscopy and the C–C/C=C-rich carbon chemical environment identified by XPS, this observation supports the formation of locally ordered carbonaceous domains with graphitic-like characteristics within an overall defect-rich tribofilm during sliding. However, carbon in the tribofilm may originate from CQDs- C_{12} , PAO4, the phenolic matrix, aramid fibers, and PTFE-related transfer species. Therefore, the combined Raman, XPS, and HRTEM evidence should be regarded as supporting local graphitic-like ordering within the multicomponent tribofilm. These locally ordered carbonaceous domains may facilitate local shear accommodation and thereby contribute to friction reduction.

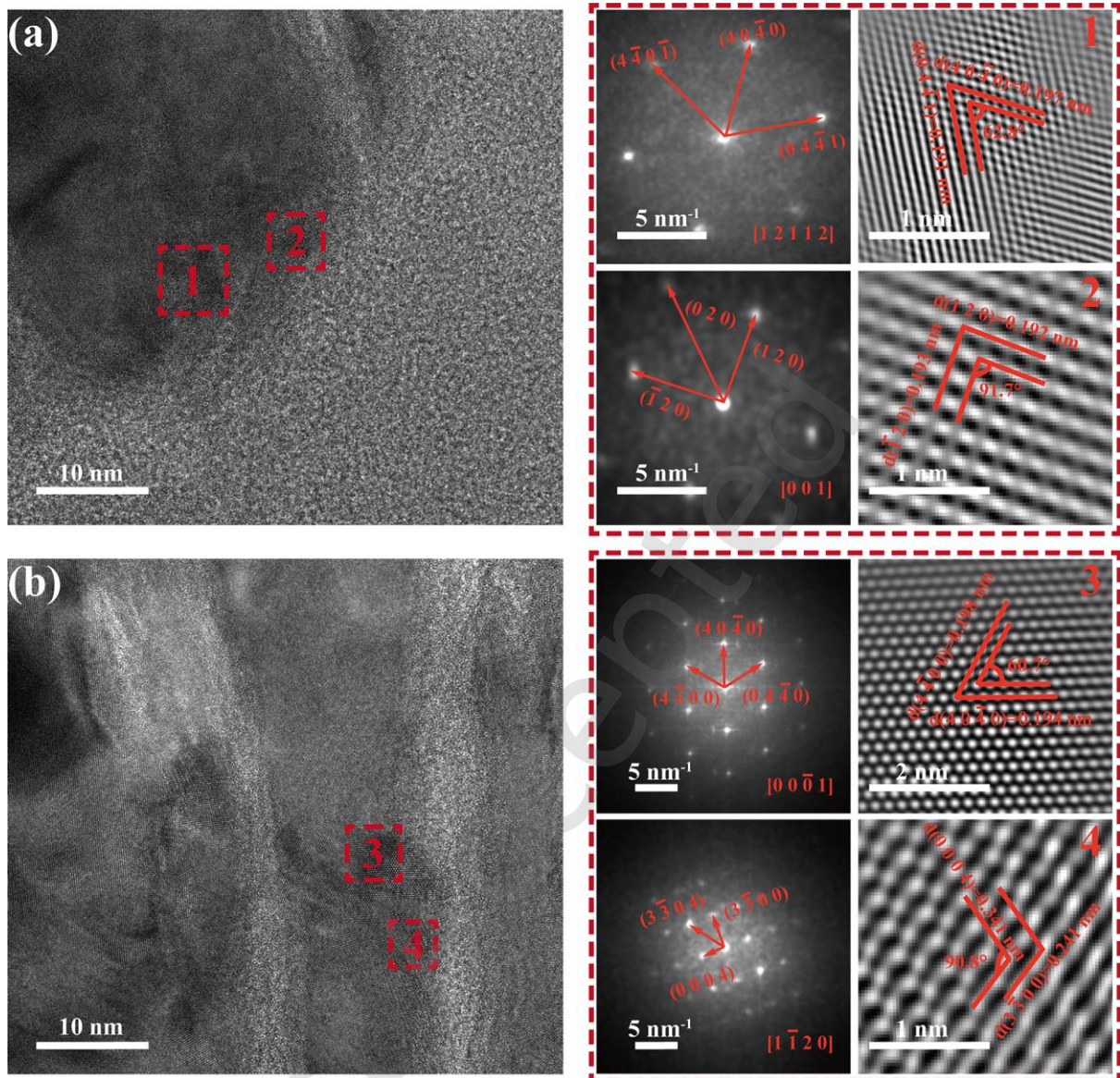


Fig. 16. HRTEM and FFT analysis of locally ordered carbonaceous domains within the multicomponent tribofilm. (a, b) HRTEM images of selected regions in the tribofilm. Enlarged images and FFT patterns of regions 1-4 reveal locally ordered carbonaceous domains and graphitic-layer-like fringes.

3.5. Mechanistic framework for optically self-reporting and self-lubricating behavior under stress

To further examine the energetic feasibility of the local graphitic-like ordering observed by HRTEM, DFT calculations were performed using a reduced active-domain model (Fig. 17).

The calculations were not intended to reproduce the entire multicomponent tribofilm or the full dynamic shear process. Instead, two compositionally identical $C_{24}H_{10}O_2$ sp^2 -rich carbon fragments were used to represent locally exposed carbon domains after CQDs-C12 enters the contact region and its surface alkyl shielding is rearranged, worn, or partially degraded. The initial configuration consisted of two non-parallel carbon domains with an interdomain tilt of approximately 42° , whereas the final configuration represented parallel graphitic-like stacking with an interplane separation of 3.41 Å (Fig. 17a-b).

To further illustrate the geometric evolution from tilted contact to parallel registry, representative structures along the seven-point constrained ordering coordinate are shown in Fig. S10. At nodes 0, 2, 4, and 6, the interdomain angle decreases from approximately 42° to 28° , 14° , and 0° , respectively, while the fitted plane separation decreases from approximately 6.33 to 3.41 Å. This structural sequence describes the approach, rotation, and noncovalent stacking of two locally exposed sp^2 -rich carbon domains rather than whole-particle graphitization of an intact CQD. The parallel stacked state was 0.745 eV lower in energy than the tilted reference state, indicating that local parallel stacking is energetically favorable within the present reduced model (Fig. 17c). A seven-point constrained ordering coordinate, incorporating simultaneous changes in interdomain tilt, normal separation, and lateral registry, continuously connected the tilted and parallel configurations. The final point was 0.794 eV lower than the initial point (Fig. 17d). The first constrained intermediate exhibited only a small apparent energy increase of approximately 0.037 eV; however, because this is a discretized constrained static-energy path rather than a nudged-elastic-band calculation, this value is not interpreted as an activation barrier. The normal-approach scan further showed that the lowest-

energy sampled separation occurred at 3.41 Å (**Fig. S11**), in agreement with the 0.341 nm graphitic-like spacing observed by HRTEM. Throughout the constrained pathway, the minimum cross-domain C-C distance remained above 3.0 Å, excluding artificial interdomain covalent-bond formation as the origin of the calculated stabilization.

After formation of the parallel configuration, a rigid one-dimensional lateral registry scan gave an energy corrugation of approximately 84 meV (**Fig. 17e** and **Fig. S12**). This relatively small corrugation supports facile local sliding between the stacked carbon domains, although it should not be interpreted as the macroscopic friction barrier of the complete tribological interface. The difference PDOS further revealed redistribution of the C-2p and O-2p contributions near the Fermi level upon parallel stacking (**Fig. 17f** and **Fig. S13**), providing complementary electronic-structure evidence for the structural rearrangement. Together with the Raman, XPS, and HRTEM observations, these calculations support the energetic plausibility of stress-assisted local sp^2 ordering and graphitic-like stacking within carbonaceous regions of the multicomponent tribofilm.

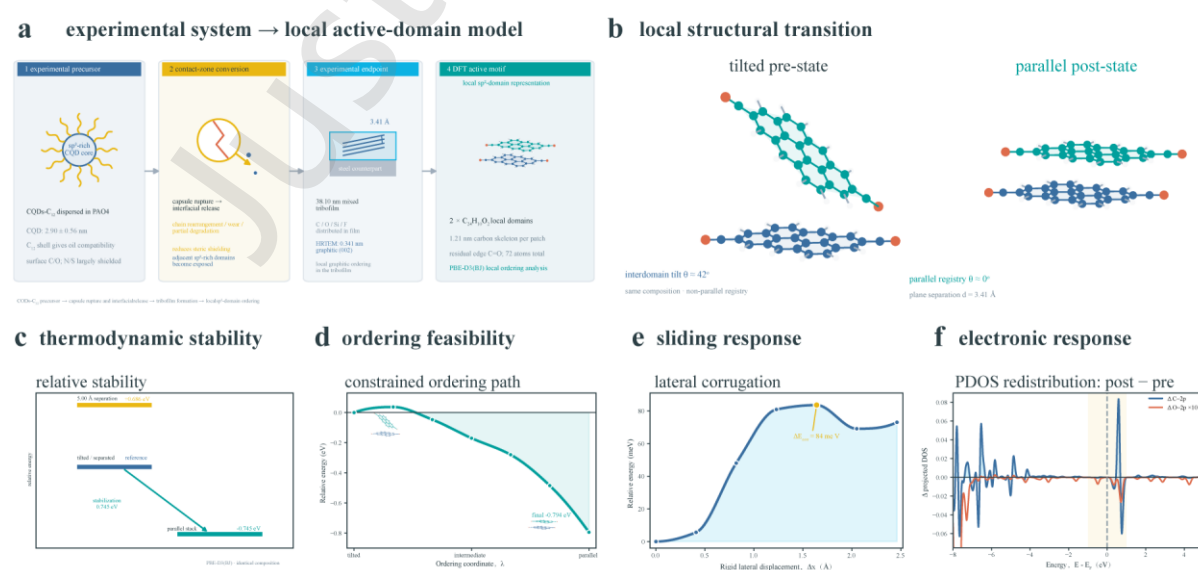


Fig. 17. DFT analysis of stress-assisted local ordering of CQD-derived sp^2 -rich carbon domains.

(a) Mapping from the experimental CQDs-C12/PAO4 system and tribological contact to the reduced local active-domain model. (b) Representative structural transition from a tilted/non-parallel pre-state to a parallel graphitic-like post-state. (c) Relative stability of the tilted and parallel configurations. (d) Relative energy along the seven-point constrained ordering coordinate (e) One-dimensional rigid lateral registry scan of the parallel stacked configuration (f) Difference PDOS (post-state minus pre-state) showing redistribution of C-2p and O-2p contributions upon local ordering.

Figure 18 summarizes the self-reporting and self-lubricating mechanism of CD-MCs/Fabric. Dodecyl-chain grafting converts hydrophilic CQDs into lipophilic CQDs-C₁₂, improving their compatibility with nonpolar PAO4 while retaining their fluorescence. This enables CQDs-C₁₂ to be dispersed in PAO4 and encapsulated in SiO₂-based microcapsules as fluorescent lubricant components.

During sliding, local compressive and shear stresses cause rupture of the embedded CD-MCs and release CQDs-C₁₂/PAO4 into the steel-ball/fabric contact region. The time-dependent increase in fluorescence provides an optical indication of the release and accumulation of the fluorescent lubricant phase. PAO4 contributes to boundary lubrication and reduces direct asperity contact at the sliding interface.

Simultaneously, transferred PTFE/AF species, SiO₂-shell fragments, and carbonaceous/lubricant-derived species are mechanically mixed and deposited on the steel counterpart, resulting in the formation of a continuous multicomponent tribofilm, as supported by the SEM/EDS and cross-sectional FIB-TEM results. The tribofilm partially separates the steel counterpart from the fabric composite and reduces direct interfacial contact and shear.

HRTEM observations reveal locally ordered graphitic-like fringes within some carbonaceous regions. These observations indicate local structural ordering of the carbonaceous species but, by themselves, do not establish extensive graphitization.

Overall, CD-MCs/Fabric exhibits a coupled self-reporting and self-lubricating behavior. Stress-induced microcapsule rupture releases the fluorescent CQDs-C₁₂/PAO4 phase, allowing lubricant delivery and accumulation to be monitored optically. Meanwhile, PAO4-assisted boundary lubrication and the formation of a multicomponent tribofilm jointly reduce direct steel/fabric contact, interfacial shear, and wear.

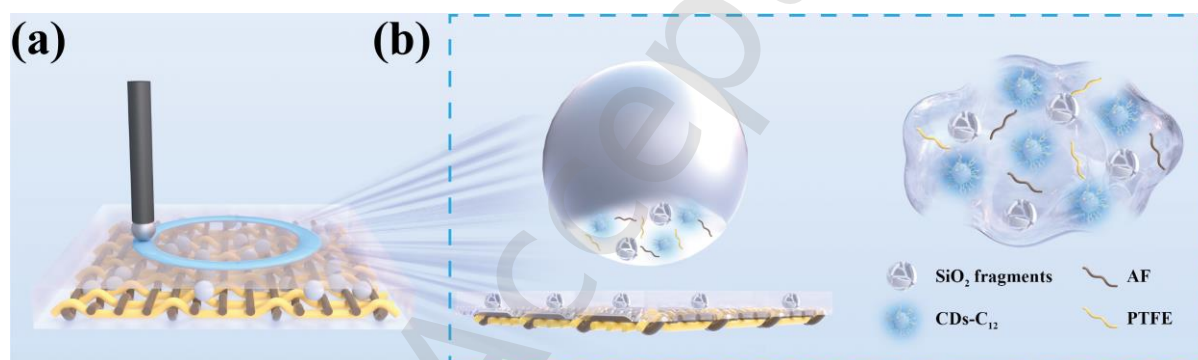


Fig. 18. Schematic illustration of the proposed optically self-reporting and self-lubricating mechanism of CD-MCs/Fabric during ball-on-disk sliding. (a) Local compressive and shear stresses induce rupture of the embedded CD-MCs and release of the fluorescent CQDs-C₁₂/PAO4 phase. (b) PAO4-assisted boundary lubrication and formation of a multicomponent tribofilm containing transferred PTFE/AF species, SiO₂-shell fragments, and carbonaceous/lubricant-derived species on the steel counterpart.

4. Conclusion

In this work, an optically self-reporting lubricant-reservoir system was developed for AF/PTFE fabric composites by encapsulating CQDs-C₁₂/PAO4 in SiO₂-based microcapsules. Dodecyl-chain grafting converted hydrophilic CQDs into oil-compatible fluorescent additives,

enabling the PAO4-loaded microcapsules to act simultaneously as lubricant reservoirs and optical probes for release.

During ball-on-disk sliding, contact-stress-induced rupture of the embedded microcapsules released CQDs–C₁₂/PAO4 into the fabric/steel contact region. The progressively enhanced fluorescence signal in the wear track provided an optical readout of lubricant release and accumulation. The optimized CD–MCs/Fabric composite achieved a friction coefficient of approximately 0.085 and a wear volume of approximately $1.03 \times 10^{-10} \text{ m}^3$, corresponding to reductions of 61.7% and 44.5% compared with Pure Fabric, respectively.

The friction and wear reduction were associated with the formation of a compact and continuous multi-component tribofilm on the steel counterpart. Cross-sectional FIB-TEM showed a continuous tribofilm with a thickness of about 38.10 nm, while EDS mapping confirmed the coexistence of C, O, Si, and F within the interfacial layer. HRTEM/FFT analysis further revealed locally ordered carbonaceous domains, including graphitic-layer-like fringes with a lattice spacing of 0.341 nm. These results indicate that released PAO4, PTFE transfer components, SiO₂ shell fragments, and CQDs–C₁₂-derived carbonaceous species jointly contributed to interfacial protection.

Overall, CD–MCs/Fabric couples stress-triggered lubricant release, fluorescence-based release reporting, and tribofilm-mediated lubrication. This design makes lubricant delivery in buried fabric/steel sliding contacts observable while reducing direct interfacial contact and wear, providing a promising route for self-lubricating fabric composites with integrated release-reporting capability.

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Declaration of Competing Interest

The authors have no competing interests to declare that are relevant to the content of this article.

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